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MICROSTRUCTURAL AND MECHANICAL CHARACTERIZATION OF SOY-BASED POLYETHYLENE FIBERS

A Thesis
Presented to
the Graduate School of
Clemson University

In Partial Fulfillment
of the Requirements for the Degree
Master of Science
Chemical Engineering

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by
Ozgun Ozdemir
August 2012

Accepted by:
Dr. Amod A. Ogale, Committee Chair
Dr. Paul Dawson
Dr. Christopher Kitchens

ABSTRACT

Bio-composite fibers have attracted increasing attention from environmentally-conscious consumers because of their low cost and biodegradability. Soy protein isolates and other purified forms have been proposed as bio-based substitutes for pure synthetic polymers for use in textiles. However, fibers from pure soy proteins do not possess adequate properties for use as textile fibers. Therefore, this research aimed to investigate composite fibers consisting of soy and polyethylene for their microstructure and mechanical properties. Changes in properties with increasing filler content and long-term properties of fibers were systematically examined.

First, the microstructure of the fibers was investigated by optical analysis. Six different compositions of soy, compatibilizer, and polyethylene were examined: 0-0-100, 0-10-90, 20-0-80, 23-7-70, 20-20-60, and 30-20-50. The pure PE and C-PE fibers had very smooth surfaces, which became rough after soy particles were added. Without a compatibilizer, soy-PE fibers had the lumpiest surface with large soy agglomerates. The soy particles on fibers made from 23-7-70 composition had a more homogenous distribution, and better dispersion (less agglomerates) than that on other fibers. Overall, the soy agglomerates oriented along the fiber direction due to shear and extensional stresses induced during flow and draw-down.

Second, the mechanical properties of fibers were analyzed. For fibers produced from 23-7-70 composition, tensile modulus reduced from 921±85 MPa to 570±94 MPa, and ultimate strength reduced from 500±90% to 67±63 % with the addition of soy to PE. Adding soy flour to polyethylene resulted in a decrease in tensile modulus and ultimate strain. Yield stress and tensile strength did not differ significantly between the fibers having 100% PE and 50% PE. However, tensile strength of 23-7-70 fibers increased to 84.2±10.6 MPa compared with that of pure PE measured at 42±6 MPa. Moreover, tensile strength of 23-7-70 fibers decreased after two month of aging. However, at ambient conditions, another four months of aging had no further effect. Overall, it changed from 84.2±10.6 MPa (non-aged) to 24.2±1.1 MPa after six months of aging at ambient conditions, whereas other mechanical and physical properties were not affected significantly. Exposure to boiling 0.1 M NaOH aqueous solution for 10 min (to simulate accelerated washing) caused a decrease in tensile strength, but additional exposure of 90 min did not cause any further reduction in tensile strength (25±4 MPa). The fiber surface became smoother due to the removal of soy particles from the surface after boiling. Thus, over-washed fibers had a lower strength at break, but the fibers retained a moderate strength. These results indicate that soy-PE fibers possess moderate mechanical, physical and long-term properties for likely use in textile applications. The primary advantage of such fiber is their lower cost relative to that of pure PE fibers.

DEDICATION

To my parents Ahmet and Tulay Ozdemir

ACKNOWLEDGMENTS

I would like to express my sincerest appreciation to my advisor, Dr. Amod Ogale, for his guidance and assistance during these two years. His skillful and scholarly guidance encouraged me to finish this thesis.

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Finally, I want to thank you to my country Turkey to support me financially during my Master‟s studies, and Mustafa Kemal Ataturk who founded the country with a secular system after a huge revolution and started to give this scholarship opportunity to us, 84 years ago.

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CHAPTER 1

INTRODUCTION

In recent decades, environmental concerns have become serious as society has become more aware of its future dependence on the environment [1]. This consciousness is leading to awareness of minimizing the use of disposable consumer goods, whose degradation time is much more than typical human age. Food grade proteins are utilized as one of the alternatives to convert purely synthetic products into partially or fully biodegradable materials [2].

Food grade proteins are natural biopolymers, which are used mostly in food products. With increasing environmental and economic concerns associated with synthetic products, they are also being used in non-food industries. Many literature studies have focused on the processing and properties of non-food products consisting of food proteins. Disposable films have been produced by using proteins and tested for their physical properties. [3]

In the last few years, biodegradability in textile industry has also become a necessity due to both economic and environmental reasons. Some of biodegradable fibers have been made of food grade proteins like corn, milk, wheat, whey, and soy. Physical properties of widely used natural fibers like silk and wool are very suitable to the textile industry, but their processing cost is relatively high. In fact, wool costs about $ 5-8 and silk $ 10-14 per pound while soy protein isolate costs about $1.20 per pound [4, 5]. In addition, animal fibers have processability problems due to variation in diameter and dyeing difficulties [6]. Thus, new kinds of textiles, containing biodegradable or

recyclable fibers, have started to become popular as world population and demand for textiles increase proportionally.

Soy flour is a cost-effective alternative ($ 0.35 per pound) as compared with other soy products such as soy isolate and concentrate, and may be a good filler or modifier for polymers, while linear low density polyethylene costs approximately $ 1.00 per pound. To date, there have not been any studies published on the mechanical properties of fibers that are blends of soy flour and linear low density polyethylene (LLDPE). It is noted that LLDPE has moderate mechanical properties and has the advantage of being easily spinnable.

This thesis sought to investigate the physical and mechanical properties of composite fibers consisting of soy and LLDPE. The interrelationship between the mechanical properties and the composition was examined. The specific objectives were to:

i. characterize the microstructure of the composite soy-PE fibers;

ii. measure the mechanical properties of fibers as a function of the microstructure, composition and size; and

iii. investigate the variation in properties of the fibers as a function of aging

The organization of this thesis is as follows: literature review relevant to this research is presented in Chapter 2. The experimental methodology conducted for this project is described in Chapter 3. Next, the results and the discussion are presented in Chapter 4. Finally, Chapter 5 summarizes the conclusions and gives recommendations for future work.

CHAPTER 2

LITERATURE REVIEW

There have been several research studies undertaken on the mechanical properties of soy composites. In this chapter, the literature that particularly involves the composites of soy protein is reviewed. The chapter begins with the properties of materials that are the components of the fibers, namely, soy-based proteins/ flours and polyethylene interactions. Then, particulate composite materials are reviewed.

2.1 Soy Protein

Protein Products from Soybean Seeds

Soybean seeds are processed by dehulling, defatting, extraction, centrifugation, and precipitation of soy beans. Three soy products are produced after seeds are dehulled and defatted: soy flour, isolates and concentrates. Soy flour in a powder form is available in four types: defatted, full fat, low fat and lecithinated. Defatted soy flour processed from dehulled and defatted flakes contains about 59% protein and less than 1% oil. Full fat and low fat soy flour are processed from defatted soy with a protein content of about 46% by adjusting the fat content to a minimum value of 18% and 4.5%, respectively [7].

Soy Protein Properties

Soy protein has peptide bonds called also amide bond. A peptide bond is the covalent bond between a carboxyl group and an amino group that is formed by a reaction mechanism as shown in Figure 2-1 [8].

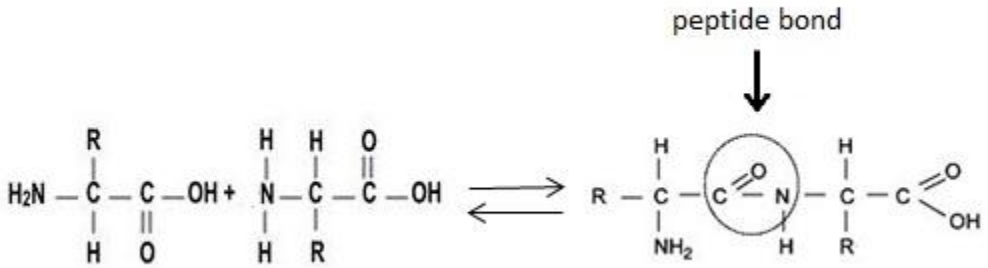


Figure 2- 1 Reaction mechanisms that produces peptide bond [8]

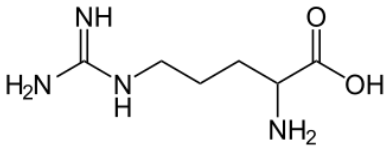
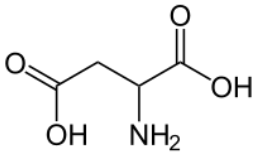
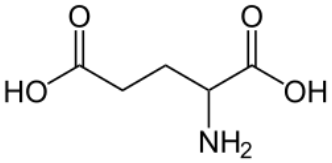
Soy protein is composed of mostly globulins whose isoelectric point is 4.5 [9]. This means that it can be dissolved in water or in dilute salt solution if the pH of the medium is not equal to its isoelectric point. Solubility of globulins is affected by pH and salts. Native state of soybean protein cannot be spun into fibers; it must first be denatured [10]. Its denaturation occurs with heat, enzymes, or alkali, which changes the protein structure from quaternary assembled subunits into unfolded structure connected by polypeptide bonds. In a heat denaturation study, it was detected that heat caused aggregates formation. After heating, proteins having low molecular weight fractions were converted to one having high molecular weight. Moreover, soybean globulins have the gelation ability when it is dissolved in a NaOH solution with more than 14.5% protein concentration [7].

Soy protein has different type of globulins classified by sedimentation coefficient 2S, 7S, 11S and 15S [11]. Glycinin and β-conglycinin content of soybean were measured respectively around 51% and 18.5% by Resurreccion et al. [12]. Thus, a major

component of soy protein is glycinin that belongs to legumin-like 11S storage globulin family, whereas the other one is β-conglycinin that is from vicilin-like 7S storage globulin family [11].

7S Globulins, the major globulin in soy protein, have three major fractions: β-conglycinin, γ-conglycinin, and basic 7S globulin. β-conglycinin is the most abundant fraction among these three. It is 30-50% of whole seed proteins where the content of γ-conglycinin and basic 7S globulin is a few percent. β-conglycinin is a glycoprotein that does not have disulphide linkage but has hydrogen bonding and hydrophobic interactions among its subunits [13]. Furthermore, glycinin has subunits consisting of both acidic and basic polypeptide chains. It is a hexamer formed by six acidic and basic subunits [14]. Glycinin has 48 sulphur atoms [11]. Soy protein has three types of amino acids: acidic (aspartic acid), basic (glutamine) and nonpolar (arginine) [15]. These three types are represented in Table 2-1.

Table 2- 1 Most important amino acids of purified defatted soy protein [7]

Amino Acid	Chemical Structure	%
Arginine		7.55
Aspartic Acid		10.38
Glutamic Acid		18.42

2.2 Linear Low Density Polyethylene

Polyethylene is a polyolefin that is broadly classified into three types based on its density: high density polyethylene (HDPE), low density polyethylene (LDPE) and linear low density polyethylene (LLDPE). An average density is 0.96 g/cm^3 for HDPE, 0.910-0.925 g/ cm^3 for LDPE, and 0.918-0.940 g/ cm^3 for LLDPE [16]. In this study, LLDPE was chosen as the matrix of composite fibers. It is a linear polymer having a backbone C-

C bonds, but it has short branches as shown in Figure 2- 2. It is a product of the reaction between a comonomer and ethylene. Typical comonomers are propylene, butene, hexene and octane [16].

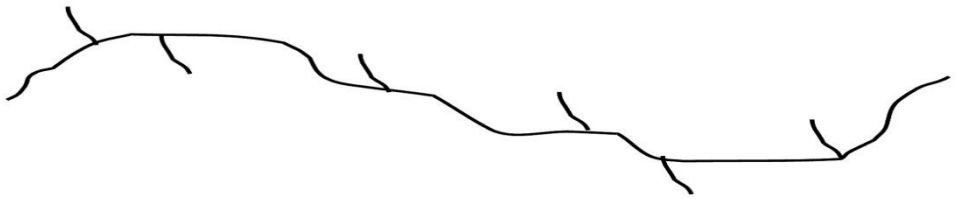


Figure 2- 1 Schematic of linear low density polyethylene molecule

LLDPE is a semi-crystalline polymer. Its melting temperature is approximately 120℃, and glass transition temperature is approximately -100℃. Polyethylene is a non-polar polymer, and therefore, not very reactive [16].This can cause difficulty of making LLDPE composites, because the bonds between LLDPE and the reinforcement phase are weak. The solution to this problem is the use of compatibilizer that can bond both with LLDPE and the reinforcement material.

2.3 Soy-based Composites

Various research studies have attempted to investigate variables, such as processing type, processing conditions, ingredients properties and the composition.

Works in recent years have been intensified on the processing soy isolate films. Cunnigham et al. [3] processed soy protein isolates with glycerol at different compositions by compression molding at 150℃. They did tensile testing on SPI fibers and found out the properties by changing plasticizer content. The films without glycerol were very brittle. When glycerol content was changed from 20 w% to 40 w%, a decrease in tensile strength and an increase in elongation at break were observed. They aged the samples at 25℃ and 50% relative humidity and tested them after 2, 3, 4, 5 and 6 weeks. Although the average tensile strength of SPI film with a composition of 20 w% was measured at 4.1 MPa, it reduced to 2.4 MPa by increasing glycerol content. Conversely, the elongation to failure rose from 1.5% to 13.2% [3].

Soy protein has low elasticity and high brittleness that comes from its strong molecular bonding, which consists of hydrogen bonding, hydrophobic interactions and polarity that makes its modulus, yield strength and tensile strength high. Addition of glycerol or methyl glycoside to soy protein makes it more flexible and processible due to the weakening interactions between protein molecules [17].

2.3.1 Soy Fibers

Fiber is defined as an elongated form of material with a maximum diameter of 250 μm and with its length to diameter ratio being at least 10. Society has used both natural and synthetic fibers. Natural ones include wool, silk, cotton, flax, whereas synthetic fibers include polyester nylon, acrylic, and elastomeric. In Table 2-2, mechanical properties of some natural fibers are presented.

Table 2-2 Properties of natural fibers [18]

Type of fiber	Density (g/cm^3)	Tensile Strength (MPa)	Young's Modulus (GPa)	Elongation at break (%)
Cotton	1.5-1.6	287-800	5.5-12.6	7-8
Flax	1.5	345-1100	27.6	2.7-3.2
Jute	1.3-1.45	393-773	13-26.5	1.16-1.5
Ramie	1.5	400-938	61.4-128	1.2-3.8

Recently, there has been a significant effort to develop new polymer fibers with better mechanical properties by making composites with fillers. In 1945, soy fibers were processed for the first time by the Ford Motor Company, and they were used for car upholstery [19]. At the same time, Huppert got a patent for chemical treatment of films and filaments. He worked on the improvement of the properties of soy protein films and fibers, which were very brittle, by applying different treatments in different chemical solutions [20]. Soybean fibers were reported with a tenacity of 0.8-1 gm/denier (80% of wool‟s strength), but they lost most of the strength in a wet state [2].

For years, soy-based fibers had not been studied much due to the lack of its wet mechanical properties, until Hammond et al. [21] studied the wet-spinning of soy protein fibers based on the study of Croston et al.[22] who described and used the method of wet-spinning and extrusion to process fibers. Hammond et al [21] processed soy fibers from

SPI by mixing it with a plasticizer and water. Primarily, they compared the mechanical properties of fibers consisting of glycerol and sorbitol and having water activity of 0.11, 0.65 and 1. An inverse relation was detected between water activity and tenacity. Also, glycerol yielded a higher tenacity and elongation at break, namely, 0.56 g/tex and 73.4%, compared to 0.33 g/tex and 1.2% for a composition having plasticizer of 15 w%.

Four different salts, $ZnCl_2$, $CaCl_2$, Na_2HPO_4 and NaCl, were tried as plasticizers at three different water activities for producing fibers by both wet-spinning and extrusion [21]. $ZnCl_2$ resulted in stronger and more flexible fibers than those resulting from other salts. Moreover, the mechanical properties of extruded soy fibers were better than wet-spun ones. Additionally, the fibers were applied a post treatment with crosslinking agents of gluteraldehyde, glyoxal, acetaldehyde and acetalanhydride. Fibers treated with glutaraldehyde and glyoxal exhibited higher tenacities than the others treated with acetaldehyde and acetalanhydride [21].

Zhang et al. [23] extruded soy fibers primarily consisting of 45 w% SPI, 15 w% glycerol, and 40 w% water by using a twin screw extruder at a screw temperature of 90℃. This soy mixture was blended with zein at different compositions such as 80:20, 70:30, and 60:40. Soy fibers, after extrusion, were treated with 1,4-benzoquinone solution, dimethylformamide solution, and dimethylsulfoxide solution at room temperature in order to improve the mechanical properties of the fibers as shown in Table 2-3. The tenacity (tensile strength) and modulus values are reported in the units of g/tex. The linear density of fibers is expressed in the units of "tex" such that 1000 m segment of 1 tex fiber weighs 1 gram.

Table 2- 3 Modulus and tenacity of soy fibers [23]

Treatment		Soy Fibers	Soy-zein Blended Fibers			Treated Soy Fibers		
			80:20	70:30	60:40	BQ	DMF	DMSO
11% rh	Tenacity (g/tex)	1.54	1.70	0.19	0.21	3.54	3.22	2.65
	Modulus (g/tex)	64	96	15	31	278	289	237
65% rh	Tenacity (g/tex)	0.55	0.44	0.15	0.18	3.37	2.53	2.93
	Modulus (g/tex)	52	17	7.1	14	280	183	190
100% rh	Tenacity (g/tex)	0.076	0.072	0.015	0.011	0.53	0.43	0.31
	Modulus (g/tex)	0.02	7.1	5.1	4.5	32	23	22

The study showed that strength of soy fibers improved most with the treatment of 1, 4-benzoquinone solution compared with the other two solutions. However, the modulus and tensile strength of soy fibers decreased at higher relative humidity. It is worth noting that the highest tenacities recorded of soy fibers were still lower than the tenacities of wool and acetate fibers. Soy fibers absorbed moisture due to its polarity; therefore, the mechanical properties were lost.

Gravier et al. [24] processed fibers from soy protein-polyester blends by extrusion. The mechanical properties of fibers decreased as soy flour content increased. For a 40 w% PET, tensile strength and elongation at break were respectively were about 4.58 MPa and 10%. They went up to 13.38 MPa and 1500% for a 70 w% PET. It had

been expected that tensile strength would increase by adding soy, but the opposite trend was observed. In addition, soy flour and biodegradable polyester blends were analyzed by SEM and it was found that soy flour particles dispersed into the matrix [24].

The blend of soy fibers and Nylon-6 (40:60) were processed through solution blowing using formic acid as the solution. The optical properties were analyzed during the study, but not the mechanical properties [25].

2.3.2 The Blends of Soy Protein and Polyolefin

In 2009, the method of preparing polypropylene/soybean protein sheets with ZnSO4 designed for an automotive interior or exterior material was patented. The tensile strength of the sheets found approximately 25 MPa [26].

Siddaramaiah et al. [27] processed LLDPE films using silica, mica and soy protein isolate as fillers with a single screw extruder. They discussed the physico-mechanical and optical properties, and the structure–property relations of soy filled LLDPE films. The LLDPE fibers containing 1 w% SPI were reported to have a tensile strength (TS) of 14 MPa and elongation at break of 144% where the pure LLDPE fibers displayed TS of 27 MPa and elongation at break of 157%. Like the other two fillers, soy caused a reduction in the tensile properties. This decrease might have resulted from the weakening intermolecular bonding of the polymer network [27].

Sam et.al [28] studied the compression molding and the mechanical properties of LLDPE - soy blends with a compatibilizer of epoxidized natural rubber. Tensile strength and elongation of the samples decreased while tensile modulus increased as soy content

increased. Pure LLDPE samples possessed a tensile modulus of 180 MPa that increased to 350 MPa after 40w% soy was added to it. This behavior was attributed to the agglomeration of soy particles within the matrix.

The changing of material properties with time is called “aging”. Aging is also observed in semi-crystalline polymers under their T_g due to their amorphous phase. However, above T_g, semi-crystalline polymers also chemically and physically are affected too [29]. The aging phenomenon of soy-PE blend samples was studied on the tensile properties of the films in another study [30]. According to the biodegradation behavior study, it was established that soy protein makes PE degradation faster, and their composites are ecofriendly [30].

From above literature studies, it is evident that there has not been a systematic evaluation of mechanical and microstructural properties of composite fibers consisting of LLDPE matrix and degradable soy-based filler. Therefore, the present study was directed towards investigating the mechanical and microstructural properties of composite fibers consisting of soy flour dispersed in a LLDPE matrix.

CHAPTER 3

EXPERIMENTAL METHODOLOGY

This chapter describes experimental procedures used to measure physical and mechanical properties of the fibers consisting of soy protein and LLDPE of different compositions. Soy flour, grade of 7B2, was provided by ADM. Linear low density polyethylene, ASPUN grade, was the matrix that was provided by Dow Chemicals. All fibers were produced in a companion study by Sam Lukubira, a Chemical Engineering PhD candidate at Clemson University.

3.1 Microstructural Analysis

Optical Microscopy

Fibers were also analyzed under an optical microscope. Paper tape was used to fix the investigated single filament on both ends on a paper tab. Fiber samples of desired size were cut randomly from the fiber roll that had been already spun. This mounting style helped further tensile analysis of fibers after their diameter was measured by optical microscopy.

Soy particle dimensions were analyzed by optical microscopy. Soy particles were spread over the microscope slide, but it was hard to see a single particle due to the agglomerates. In order to disperse the particles, several drops of acetone were put on the

agglomerate. Acetone provided the dispersion of the small agglomerates and prevented the movement of particles.

Microstructure analysis was performed with an Olympus BX60 Microscope. The microscope was operated in transmitted light mode. Single fibers mounted on paper tab were placed on a microscope slide, and the slide was placed on the microscope stage that could be translated and rotated. For a quick focus, the light circle reflecting on the sample surface was observed and the knobs were adjusted when that circle became smallest. Fine focusing was done by looking through the eye piece. The microscope is connected to a monitor and a camera that is connected to a computer. Image PRO Plus software was used that helps to digitized measured diameter of fibers and area of soy particles from their photomicrographs. Objective lenses with 10x, 20x, 50x and 100x magnifications were used in conjunction with 10x magnification of the view piece. The light level of the microscope was adjusted to more than 7/12 when capturing images.

Image Analysis

Under the optical microscopy, the soy particles on fiber surface cannot be detected well. In order to do surface analysis and get better contrast, staining process was needed. Dye solutions were originally produced and analyzed in a companion study by Chinmay Naphade, a Food Science MS student at Clemson University. The fibers were stained with Thermo Scientific Pierce Coomassie Brilliant Blue R-250. This dye has a form of disulfonated triphenylmethane. Protein color changes to purple when it binds to the dye that has a maximum absorption 595 nm (Figure 3-1).

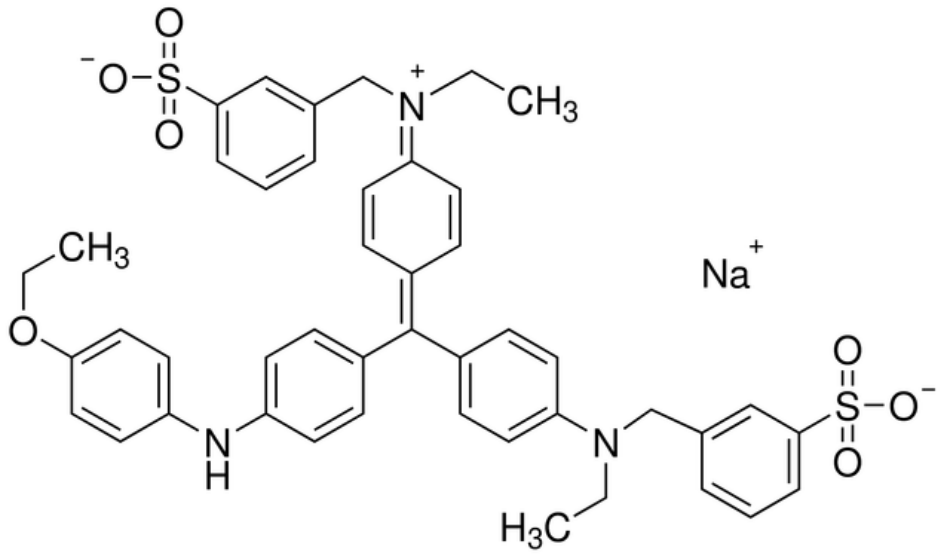


Figure 3- 1 Chemical structure of Commasie Brilliant Blue dyes R-250 [31]

Fiber staining procedure with Coomassie Brilliant Blue R-250 (B014) is as follows. First, staining solution was prepared by using 0.25 % Sigma Aldrich Coomassie Brilliant Blue R-250 (B014), 40.00 % methanol, 7.00 % acetic acid, and 52.75% deionized water. Dye was dissolved in this solution, and the fibers were dipped into the solution for about 90 min. Next, a destaining solution was prepared by 5% methanol, 7% acetic acid and 88% deionized water. The stained fibers were dipped into the destaining solution for one and a half hour.

For image analysis of dyed fibers, Photoshop 6.0 was used on some of the pictures to differentiate colors. Micrographs were cut from their axial center where the focus was better, and no shadow was formed. By Photoshop 6.0, these cut micrographs of well-focused parts were zoomed into higher sizes. Next, those cut and zoomed parts were

copied back to Image Pro plus program in order to measure the surface areas of particles. By selecting “Measure” and then “Count-size” menu items, the color range of soy particles was manually selected according to their color. Next, the program calculated the area of particles and the whole area of fiber. The particle area as a ratio of total fiber area was calculated for the different composition fibers. Orientation of soy particles on the surfaces of fibers was also determined by optical microscopy. Relative to fiber axis, the particles orientation was measured by Image Pro Plus.

3.2 Mechanical Testing

Tensile Testing Unit

Uniaxial tensile tests were conducted on an ATS 900. A 5 lb load (22.2 N) cell was used at 10 % of its capacity to test the fibers. The tests were performed in the laboratory environment at a temperature of around 298 K and relative humidity of approximately 50 %. The upper clamp of the equipment was removed and instead a hook was attached to the load cell as shown in Figure 3-2.

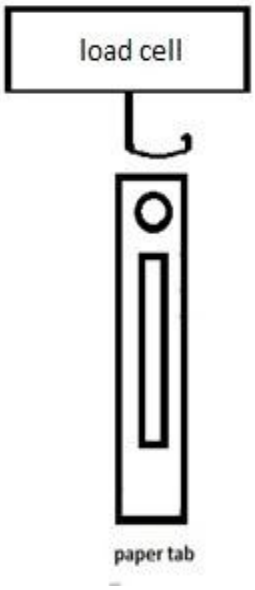


Figure 3- 2 Modified upper grip (hook) and punched paper tab

A Linseis L6012B chart recorder was attached to the ATS 900. It recorded the force-elongation curves, which allowed the calculation of tensile modulus, yield stress, elongation at yield, tensile strength and elongation at break.

Samples Preparation

Paper tabs containing 25 mm long opening in the middle were punched at the top in order to hang it on the hook attached to the load cell. Monofilaments were randomly selected from each batch of rolls using tweezers, and they were cut to a length of about 37 mm. Using an epoxy adhesive, the samples were mounted 6 mm from both ends on paper tabs that had been already pre-cut (Figure 3-3).

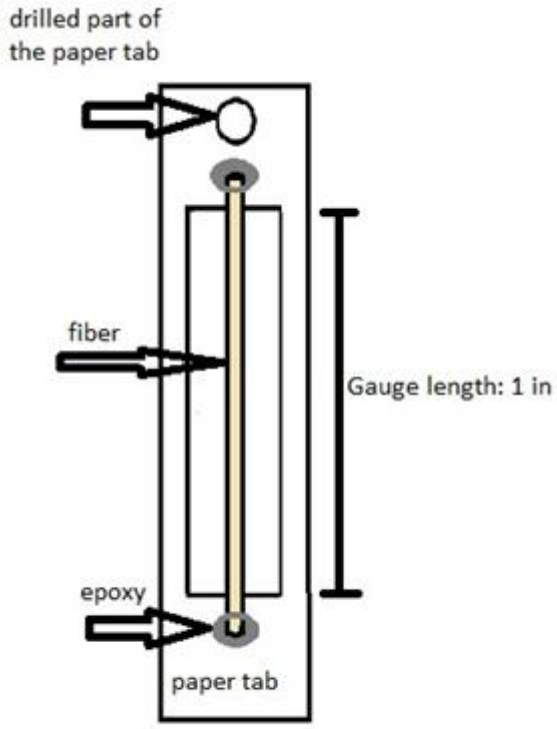


Figure 3- 3 Schematic of a single fiber mounted on a paper tab

Before the application of epoxy, the fibers were aligned parallel to the paper tab with small pieces of paper tape. Then, epoxy and hardener were mixed properly. Small droplets of this mixture were applied to fibers ends. The samples were placed in the laboratory during the curing of epoxy for a minimum of 3 hours.

Tensile Testing

The aim of tensile testing was to measure Young‟s modulus, stress and strain at yield, and stress and strain at break. Young‟s Modulus is the initial slope of the stress-

strain curve, and characterizes the stiffness of the fiber in the elastic region of a material. Stress is the internal force per cross-sectional area. Strain is defined as the relative change in size of the sample due to the applied force.

Stress= $\sigma = F / A_0$

Strain= $\epsilon = \Delta L / L_0$

Tensile Modulus= $E = \sigma / \epsilon$

where F is force, A_0 is cross-sectional area of the sample, L_0 is initial length of the sample.

A representative stress strain curve for a ductile material together with material properties that can be determined from various part of the measured curve is shown in Figure 3-4.

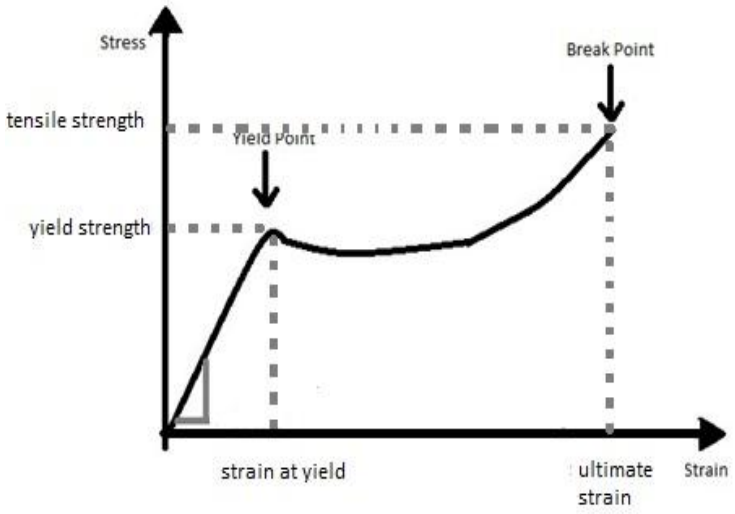


Figure 3- 4 Representative stress- strain curve of a ductile material

ASTM D2256/D2256M Standard Test Method for Tensile Properties of Yarns by the Single-Strand Method was primarily followed for the tests. Some modifications in the standard procedure were done due to equipment limitations. A crosshead speed of 2 in/min was used on a sample gauge length of 1 inch (instead of the standard crosshead speed of 10 in/min for a sample gauge length of 10 in). Moreover, one of the clamps was removed from the ATS 900, and a hook was attached instead of the clamp. The ASTM procedure suggests forming knots when using a hook. Knotting was tried, but the adjustment of the sample gauge length was very hard by knotting. Therefore, paper tab was used instead. At least ten replicates were tested for each fiber type. For limited batches, there were 5 replicates were tested.

The diameter of 25 mm long fibers was measured at nine points along its length by using an optical microscope (detailed in Section 3.3) to calculate an average diameter. The sample was hung from the hole in the paper tab on to the hook of the load cell. The lower grip was adjusted to match the gauge length of the sample attached to the paper tab. After the grip was closed, the force on the sample was zeroed out. The load, peak load, and displacement readout was reset. The crosshead was moved downward at a rate of 2 in/min (5.08 cm/min). The force-elongation data was recorded on the strip chart recorder.

The primary compositions of the fibers tested were:

a) 30 w% soy-20 w% C-50 w% PE (30-20-50)

b) 20 w% soy-20 w% C -60 w% PE (20-20-60)

c) 23 w% soy-7 w% C -70 w% PE (23-7-70 or soy-C-PE)

d) 20 w% soy-0 w% C-80 w% PE (20-80 or soy-PE)

e) 0 w% soy-9 w% C-91 w% PE (C-PE)

where „soy" stands for soy flour (7B2), C for a compatibilizer, and PE for Dowlex LLDPE. A majority of fibers tested had an average diameter of 55 μm. However, fibers with different diameters (45-55-65 μm) made from pure PE composites were also tested to establish a relationship between the diameter and mechanical properties of soy-C fibers.

Long-term Properties

An accelerated washing simulation was carried out by immersing fibers in boiling NaOH aqueous solution. Pure polyethylene and soy-C-PE fibers were boiled for 10 min and 100 min in 0.1 M NaOH solution. Tensile testing was conducted on boiled samples, and the results were compared with those from non-boiled ones. In another set of experiments, samples were aged under ambient conditions to assess the effect of long-term storage to determine whether the mechanical properties change with storage at ambient conditions. Fibers were aged in the laboratory environment at a temperature of around 298 K and relative humidity of approximately 50 %. Tensile tests were conducted on the aged fibers for comparison with non-aged ones.

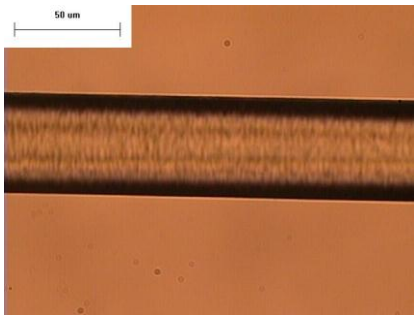
CHAPTER 4

RESULTS AND DISCUSSION

This chapter presents microstructural results obtained from optical microscopy and tensile test results obtained from various types of fibers tested. The examined mechanical properties of specimens were elastic modulus, yield strength, yield strain, tensile strength and break strain. The fiber mechanical properties were examined based on their polyethylene content. Fibers having a nominal diameter of 55 μm were compared. The effect of fiber diameter and aging conditions on the tensile properties was also investigated.

4.1 Microstructural Characterization

Pure PE fibers Figure 4-1(a) is a typical optical micrograph of the pure PE fiber. As expected, PE fibers had a plain lateral surface, and the diameter is fairly constant at approximately 55 μm. The fibers were dyed as a control for the further image analysis of soy fibers. However, as seen in Figure 4-1(b), no significant color change can be observed, because the dye does not bind to nonpolar polyethylene.

a) 

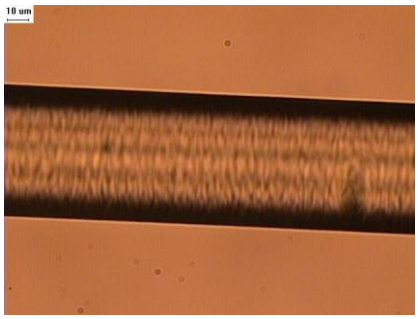
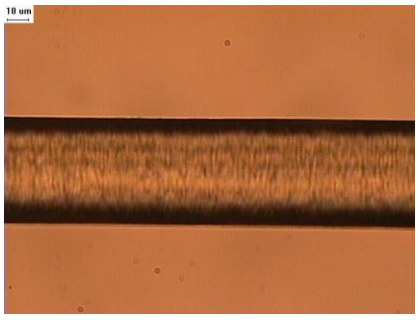
b) 

Figure 4- 1 Optical Micrograph of (a) PE fiber and (b) dyed pure PE

PE-C Fibers

Figure 4- 2(a) exhibits a representative micrograph of PE-C monofilament, which displays a smooth surface and a consistent diameter. It looks similar to pure PE under the optical microscope. For control purpose, these fibers were also dyed, and the micrograph is displayed in Figure 4-2(b). Again, the dyed PE-C fibers did not show any major color change.

a) 

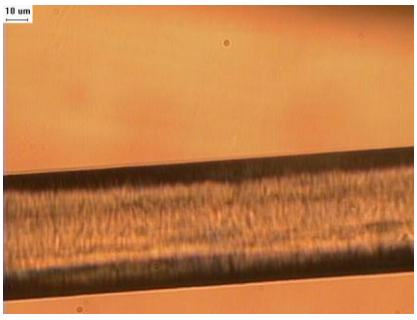
b) 

Figure 4- 2 Micrograph of (a) C-PE and (b) dyed C-PE

Soy Flour

The diameter of soy flour particles was first measured by analyzing optical micrographs such as that shown in Figure 4-3. The cross-section appeared nominally circular, so it was inferred that the particles were nominally spherical. The average projected cross-sectional area of particles was measured to be 9.1 ±2.3 μm^2. Therefore, their diameter was calculated as nominally being 3.4±1.7 μm.

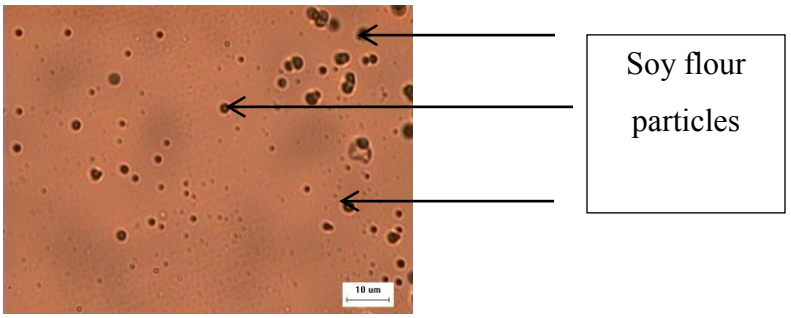


Figure 4- 1 Optical micrograph of 7B2 soy flour

Soy-PE Fibers

Representative optical micrographs of soy-PE fibers are shown in Figure 4-4 (a) and (b). The dark colors represent dense soy components. To measure the soy content on the surface, fibers were dyed. Figure 4-4(c) and (d) are optical images of dyed fibers obtained under two different magnifications where the purple areas represent soy particles.

In Figure 4-4(a), big particulates can be seen on the surface. Figure 4-4(b) displays a monofilament containing a soy agglomerate in the middle. As seen in the

figures, the surface of these (20-80) fibers is rough as a consequence of the soy agglomerates. There is also a significant variation in diameter of fibers. The diameter was 55 μm at one end of the fiber, but was 85 μm at the other end (Figure 4-4 (a)). Diameter varies from 35 μm to 100 μm for the fiber displayed in Figure 4-4(b). The agglomerate can be seen more clearly in Figure 4-4 (c), and the micrographs confirm the presence of large agglomerates (top right corner).

By image analysis (Image Pro), it was determined that there were on the average 554 ±120 protruding particles per mm2 of fiber surface area. The particle count should have been much higher than this, if all particles had been dispersed individually. An average particle cross-sectional area was calculated as 602±322 μm2, which was significantly higher than a single particle area (9.1±2.3 μm2). The orientation of soy agglomerates on the fiber surface was generally in the fiber axis direction. This is consistent with the fact that the shear stresses encountered in the spinneret and the extensional stresses exerted during fiber draw-down tend to align noncircular particles in the flow direction, which is the fiber axis direction. In Figure 4-4 (d), while a mass of soy agglomerate exists with a diameter of approximately 15 μm shown in the first box, the second box has two single particles, showing that there is no homogeneity in the agglomerate size.

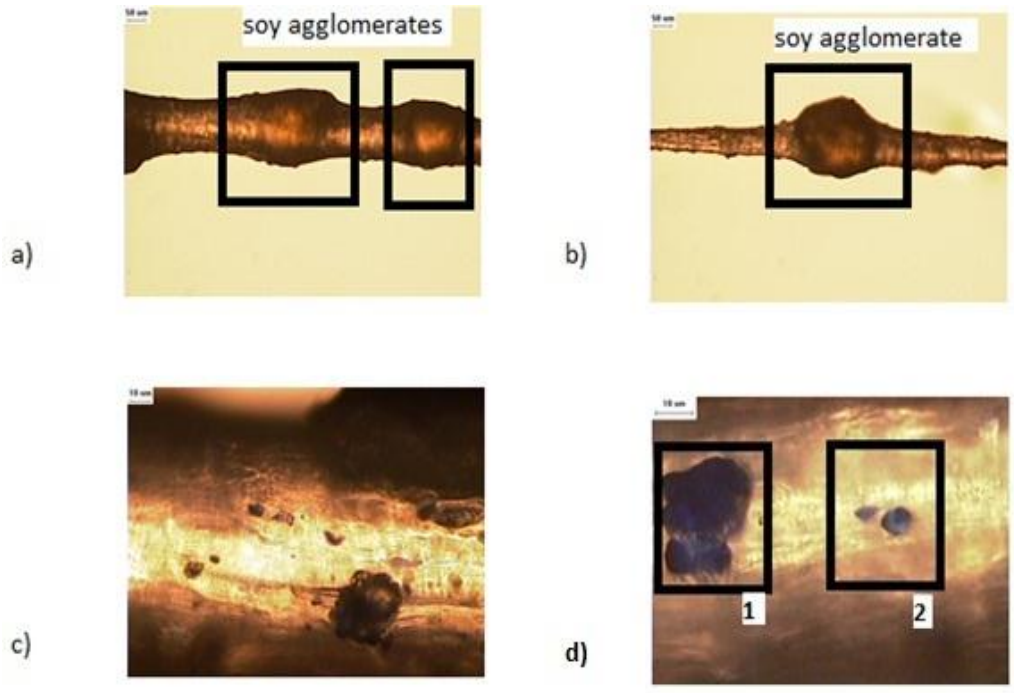


Figure 4- 2 Optical micrographs of 20-80 fibers: (a) non-treated under 50 x, (b) non-treated under 100 x magnification, (c) dyed under 50x, and (d) dyed under 100x magnification

By image analysis, the soy content on the fiber surface was found to be 16 ±3.4 % (remainder PE). This data is generally consistent with the soy/PE content ratio of 20/80 by weight, which corresponds to 14/86 by volume. Therefore, there is no significant accumulation or depletion of soy flour on the surface as compared with the interior of the fiber.

A representative optical micrograph of fibers is displayed in Figure 4-5(a) for a blend containing 30-20-50 content of soy-C-PE. The lighter parts of this micrograph represent polyethylene-rich sections, and darker areas are soy-rich sections. However, the microstructural details were not very clearly seen for such unstained sample. To detect

the soy particles more clearly, the fibers were dyed with Commassie blue staining agent. Figure 4- 5(b) displays a lateral section of a dyed fiber. The purple areas on the micrographs are soy particles as the dye preferentially stains the proteins contained in soy. The darker edges are due to the fact that the fiber was cylindrical and the microscope was not able to focus on its edges, when focused on its middle section.

The image analysis results showed that the average diameter of particles on the fiber surface was 18.8±12.5 μm. Additionally, the protruding number of agglomerates in one mm2 area was detected as 871 ±419. It is interesting to note that the cross-section area of some "purple" particles is much more than the area of an average particle calculated before, and some particles do not seem spherical. Those big areas do not belong to single particles, but to agglomerates. In fact, the roughness of the fiber surface seen in the Figure 4-5 (a) and (b) is due to the accumulation of the particles into a bigger lump, because particles did not disperse in the matrix thoroughly. Rather, they gathered and possibly created new agglomerates during processing. Therefore, the number of particles seems less than its actual number seen for measurement of pure soy flour (Figure 4- 3). Most of these agglomerates elongated along the fiber axis direction.

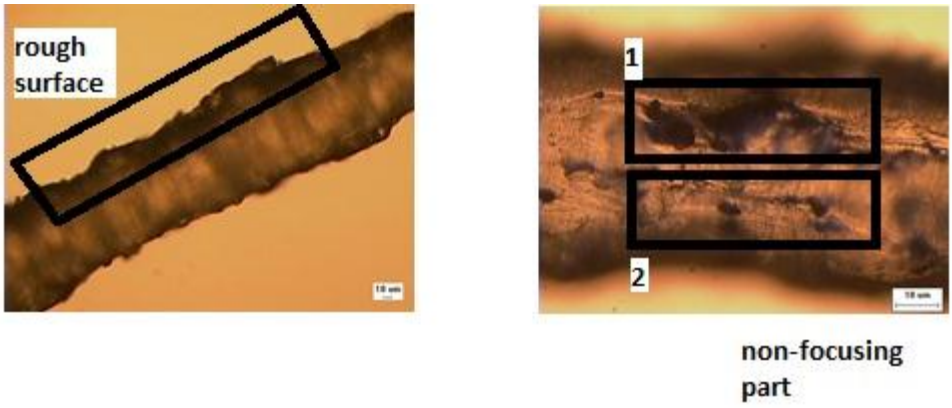


Figure 4- 5 Lateral section of a 30-20-50 fiber (a) no treatment and (b) dyed

By image analysis, the average area of soy particles on the fiber surface was calculated as 16 ±5 % of the total fiber area. However, based on the overall soy content added during processing, 21.4% by volume of this fiber is soy flour. This suggests that the interior of the fiber has slightly higher soy content than its overall content, indicating that the particles were not dispersed homogenously in the fiber. In Figure 4-5(b), the distribution of particles on a monofilament is shown. While the cross-section labeled "1" has big soy agglomerate with a higher area, the cross-section labeled "2" has less soy area, showing again that particles were not dispersed uniformly on the fiber surface.

Next, more PE was added to the blend to get fibers having a composition of 20 w% soy, 20 w% compatibilizer and 60 w% PE. A representative optical micrograph of this fiber is displayed in Figure 4-6 (a). The lighter area of the image represents polyethylene-rich phase. To differentiate between polyethylene and soy sections more clearly, fibers were again dyed. Figure 4-6 (b) displays a lateral section of a dyed fiber where the purple areas on the micrographs represent the soy flour. Image analysis showed

that the average protruded particle area on the fiber surface is between 107 and 69 μm^2. This value is approximately ten times that of an individual particle area of 9.1 ±2.3 μm^2, again pointing to the existence of agglomerates on the fiber surface. One of them can be seen in the first section of Figure 4-6 (b).

Furthermore, it was observed that the fibers do not have a smooth surface (Figure 4-6 (a) and (b)) due to the agglomeration of the particles on the surface. The number of agglomerates in one mm2 area were counted as 1438 ±165. If all the fibers had been present as individual particles, then their count would have been an order magnitude larger. The agglomerates were generally oriented along the fiber axis direction. By image analysis, the nominal soy content on the fiber surface was calculated as 16 ±5 % (based on the total fiber area), which was similar to the overall composition of 14 vol% added in the original blend. In Figure 4-6 (b), from a comparison of the first and second sections, it can be inferred that the content of soy in the first area is higher than that in percentage of the second one. Again, this confirms that there is not a homogenous dispersion of particles in the fiber.

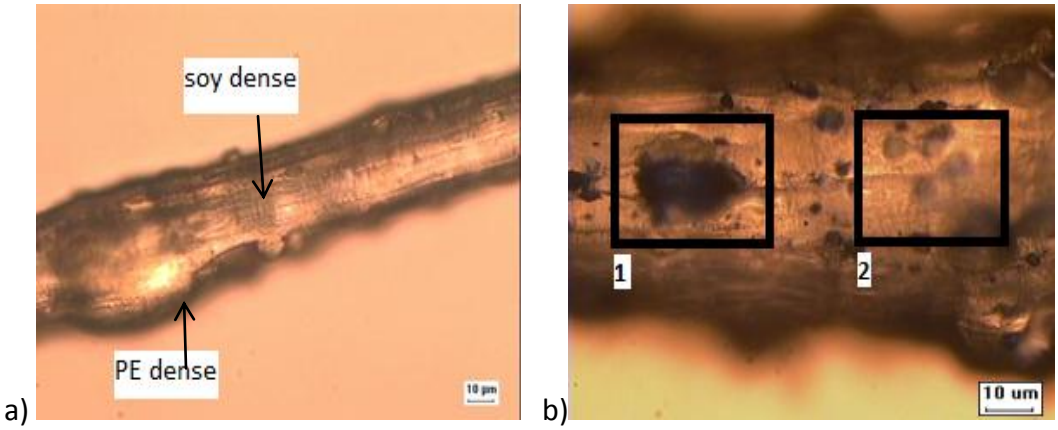


Figure 4- 6 Micrograph of a 20-20-60 monofilament: (a) no treatment and (b) dyed

Finally, a representative optical micrograph of fibers produced for 23-7-70 composition of soy-C-PE is displayed in Figure 4-7 (a). Several soy particles (circled) could be detected from their dark color on the image. To see polyethylene and soy sections more clearly, fibers were again dyed, and Figure 4-7 (b) displays a lateral section.

The image analysis results showed that soy agglomerate area on the fiber surface ranged between 75 and 133 μm^2, indicating an average particle diameter of 11.1±6.1 μm. On the fiber, both single particles and small agglomerates exist as seen in Figure 4-7 (b). There are agglomerates, but their size is not as big as those seen for 20-80 or 30-20-50 compositions (Figure 4-4 and Figure 4-5). The number of agglomerates in one mm2 area was calculated as 1655 ±254 for 23-7-70 composition fibers. On a normalized basis, this would be equivalent to approximately 1439 agglomerates/ mm^2 if soy content were 20% (like that in 20-20-60 fibers). Although, the dispersion of particles is still not

homogenous, from Figure 4-7 (a), it can be concluded that this type of fiber had a better dispersion as compared to the other three types of fibers. Another relative difference observed was that 23-7-70 fibers had smoother surface, as shown in Figure 4-6 and 4.7, when compared with the earlier fibers.

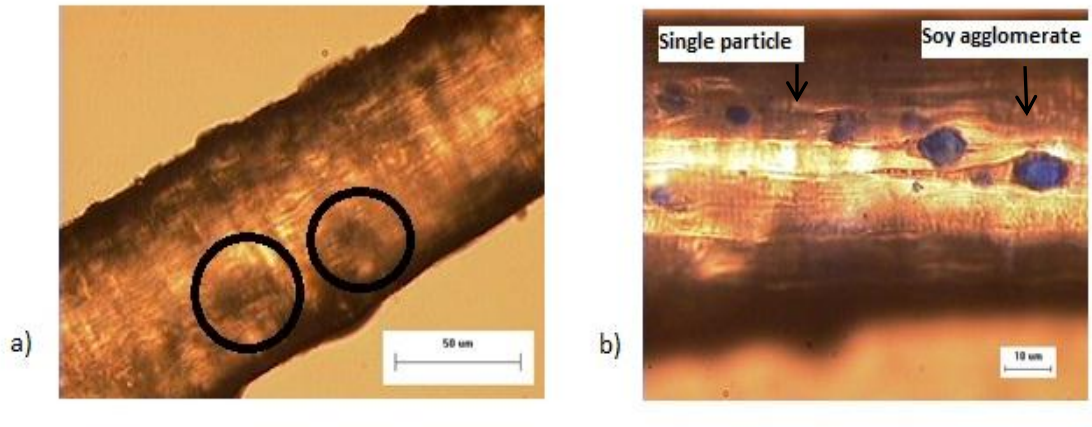


Figure 4-7 Optical micrograph of 23-7-70 monofilament: (a) no-treatment and (b) dyed

The bulk composition of this fiber is 16 vol% soy, 9 vol% compatibilizer and 75 vol% PE by volume. It was observed that 15.9 ±3.4% of fiber surface area consisted of soy particles. Thus, the particles were not accumulated in the interior or the surface of the composite fibers.

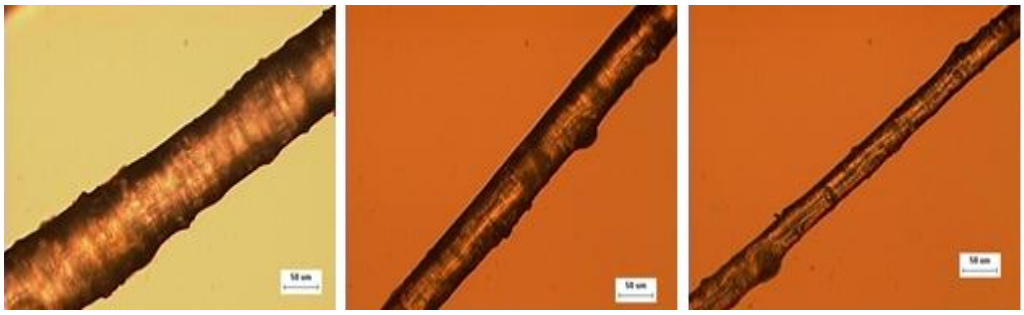


Figure 4- 8 Optical micrographs representing the 23-7-70 fibers with different diameters

Figure 4-8 displays the fiber images with different diameters that were produced to test the effect of the diameter on mechanical properties. No significant difference could be detected in the overall microstructure of these fibers relative to the ones having an average diameter of 55 µm .

It is known that the spherical particles cannot have any orientation, but the agglomerates had elliptical shape and they were analyzed for orientation. Based on the results presented in Table 4-1, agglomerates were mostly oriented at 0-10° or 170-180° relative to the fiber axis The agglomerates rarely orientated transversely (90°).

Table 4- 1 Orientation of soy agglomerates on the surface of 23-7-70 fibers

X= slope of the particle in degrees	Count	count/total (%)	X= slope of the particle in degrees	count	count/total (%)
0	25	9.2	60<x <70	2	0.7
0<x<1	14	5.2	80<x <90	0	0.0
1<x<2	9	3.3	90<x <100	0	0.0
2<x<3	13	4.8	100<x <110	1	0.4
3<x<4	4	1.5	110<x <120	1	0.4
4<x<5	6	2.2	120<x <130	2	0.7
5<x<10	33	12.2	130<x <140	3	1.1
10<x <20	45	16.6	140<x <150	5	1.8
20<x <30	16	5.9	150<x <160	10	3.7
30<x <40	2	0.7	160<x <170	18	6.6
40<x <50	2	0.7	170<x <180	59	21.8
50<x <60	2	0.7			

Long-Term Microstructure

In an effort to determine the long-term stability of the soy-based fibers, samples were aged under ambient conditions to simulate long-term storage. As displayed by a representative micrograph of Figure 4-9, no significant difference was observed in the microstructure of the aged fibers relative to that of the non-aged ones.

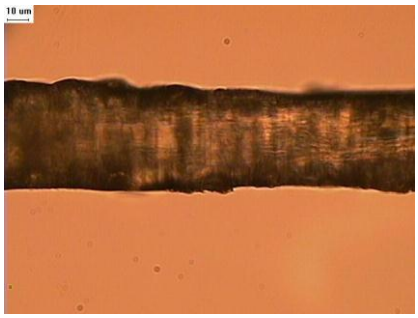


Figure 4- 9 Optical image of fibers aged at ambient conditions for 6 months

To simulate repeated washing, fibers were boiled in NaOH aqueous solution for different durations. After 10 min of immersion in boiling NaOH aqueous solution, voids were formed on the surface of the fibers, as seen in Figure 4-10. The fibers were dyed after boiling treatment, to clearly identify soy-rich areas. However, as shown in Figure 4-11, no purple areas were detected on the surface. From earlier studies, it is known that there is some soy flour on the surface, but no significant soy was detected protruding from the surface of the boiled fibers.

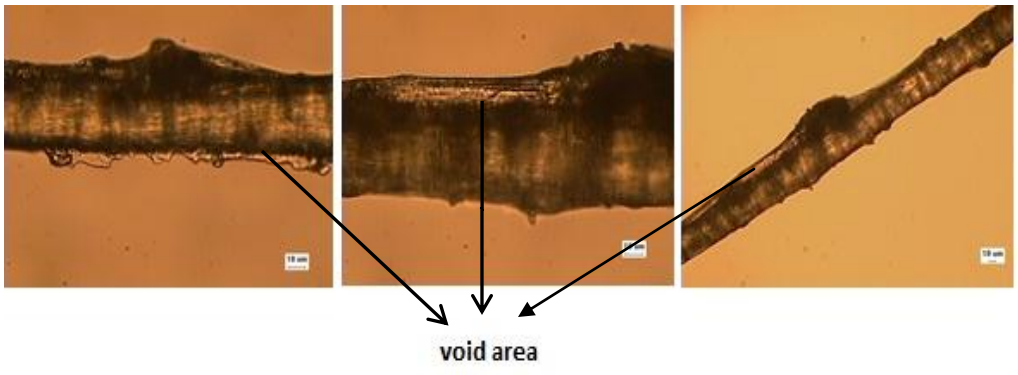


Figure 4- 10 Optical images of undyed fibers after 10 min boiling

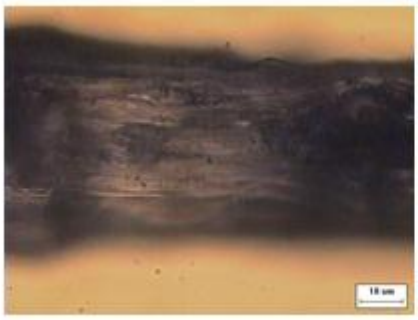
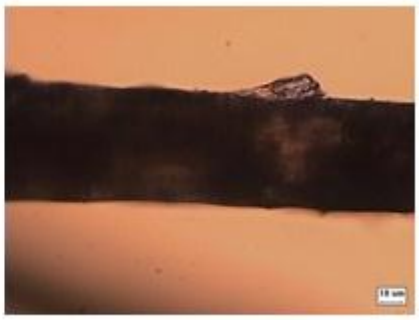
a)  b) 

Figure 4- 11 Optical images of dyed fibers after 10 min boiling taken by (a) 100x and (b) 50 x magnification

Figure 4-12 displays micrographs of fibers after 100 min boiling. The fiber surface became significantly smoother than the original fibers due to almost complete dissolution of soy particles. Some protrusions are still visible, which are likely soy particles in the interior of the fiber that are completely encapsulated by the PE matrix.

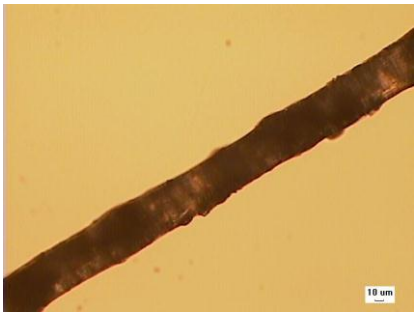
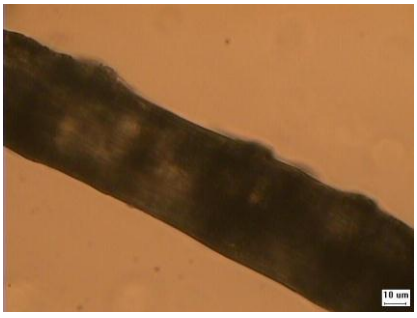
a)  b) 

Figure 4- 12 Optical micrograph of Fibers after 100 min boiling: (a) no-treatment and (b) dyed taken under different magnifications

Boiled and non-boiled pure PE fibers are presented in Figure 4-13 for comparison purpose. There is no significant difference observed from the micrographs on the surface texture or diameter uniformity.

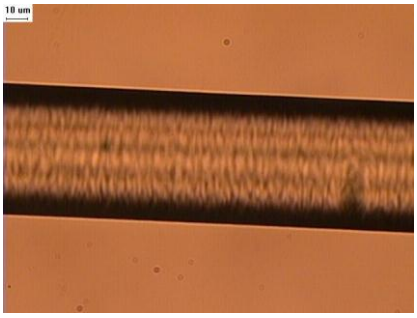
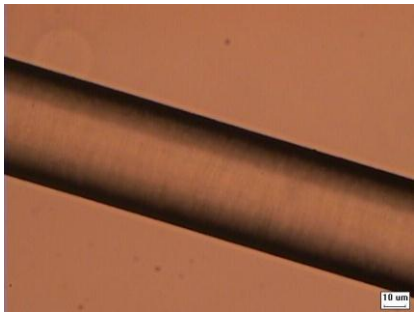
a)  b) 

Figure 4- 13 Optical micrograph of Pure PE (a) no treatment (b) boiled

4.2 Mechanical Characterization

Mechanical properties of fibers were compared according to their polyethylene content for fibers having an average diameter of 55 µm. The data were analyzed by SAS software. The multiple comparisons by Tukey‟s method in SAS were applied to do one-to-one comparisons of the means and differences [32]. The level of significance used was 5%.

Fibers Without Soy

Pure PE

A representative stress-strain curve of PE is displayed in Figure 4-14, which shows that the PE fibers are very extensible. It has a strain of 5.7±1 % at yield and 500±90 % at break. Fibers show a yield stress of 25±2 MPa and a tensile strength of 42±6 MPa. PE fibers had an average tensile modulus of 921±85 MPa.

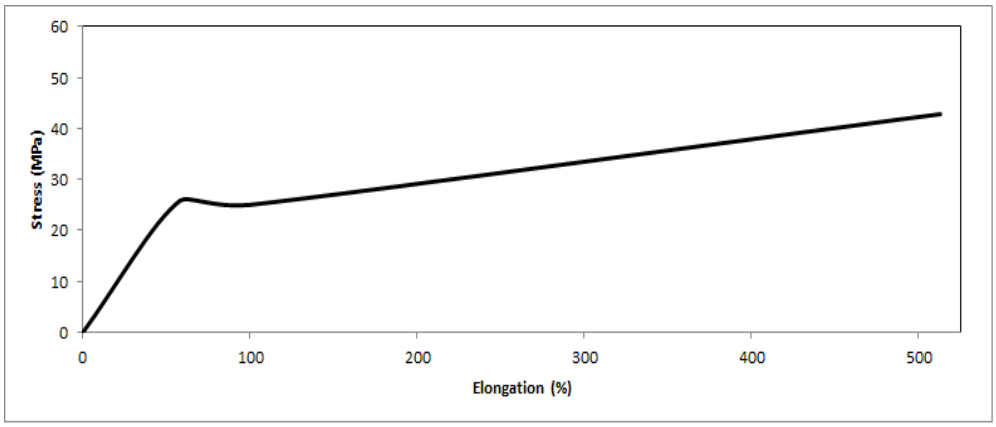


Figure 4- 14 Stress-strain curve for pure PE

PE-C

After 10 w% compatibilizer was added to pure polyethylene, the tensile modulus decreased to 638±44 MPa. There was also a decrease in yield strength from 25±2 MPa to 18±1 MPa. However, the yield strain of 4.9±0.5 % and the tensile strength of 30±2 MPa did not show a significant change relative to pure PE fibers, but the ultimate strain increased to 733.7±131.7 %. As expected adding a low molecular weight compatibilizer

to polyethylene resulted in fibers having less modulus, less strength, but more elongation. Thus, the blend was more ductile relative to pure PE.

Fibers With Soy

Soy-PE

The tensile modulus of 20-80 soy-PE fibers was 655.1± 80.2 MPa (Figure 4-16), its yield stress was 16.9±1.7 MPa (Figure 4-17), and its break strength was 28.6±3.1 MPa (Figure 4-19). The strains were 4±0.2 % at yield and 145±30.8 % at break. Compared with the ones for pure polyethylene fibers, all mechanical properties of 20-80 fibers reduced significantly.

If the soy particles had been dispersed homogenously in the PE matrix, as shown in Figure 4-15 (a), the expectation might have been an increase in the tensile modulus due to the fact that soy is a solid that has a higher modulus. However, the soy particles formed agglomerates inside the matrix (Figure 4-15 (b)). In addition, without a compatibilizer, the bonds between soy flour and PE are very weak. Also, the agglomerates themselves are very weak as they have void volume inside. For these reasons, the modulus and strength reduced, as did the strain at break. This means that the fibers became less extensible. The reason likely is that the interphase between matrix and particles is not strong enough without compatibilizer. Note that pure soy strain to failure is approximately 3 % as compared 500 % for pure PE. It was expected that soy would cause a reduction in elongation.

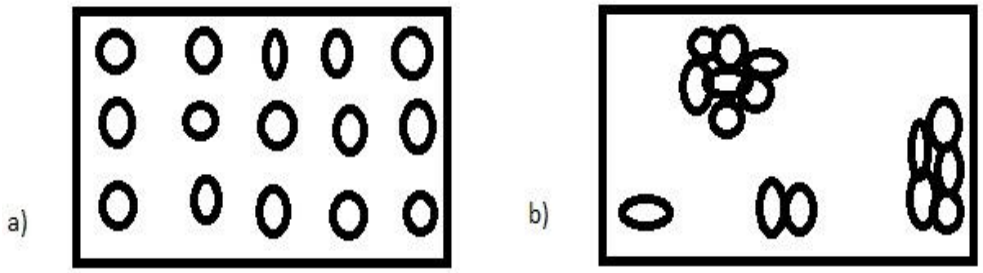


Figure 4- 15 Soy particle distribution in matrix (a) homogenously and (b) non-uniform

Soy-C-PE (30-20-50) fibers

The tensile modulus was measured at 570±94.5 MPa, which was significantly less than that of PE (921 ±85 MPa), but it was not significantly different than that of C-PE fibers (638±44 MPa) as shown in Figure 4-16. The yield stress of the fibers (29.4±11.3 MPa) was higher than that of C-PE fibers (18±1), whereas the break strength was measured at 42±8.4 MPa, which was lower than that of C-PE fibers (30±2 MPa) as seen in Figure 17 and 19. The strain was measured to be 8.5±2.3 % at yield and 68±63 % at break.

Soy-C-PE (20-20-60) fibers

Increasing the content of PE while decreasing the content of soy resulted in an insignificant difference in modulus (620.4 ±91 MPa), yield strength (18.5 ±2.7 MPa) and tensile strength (34.3 ±5 MPa) compared to those of 30-20-50 fibers. As Figure 4-18 and

20 display, the strain also did not show any significance difference, which was 7±1.3 % at yield and 251±24 % at break point.

Soy-C-PE (23-7-70) fibers

For fibers containing 23-7-70 ratio of soy, compatibilizer and PE, the tensile modulus was 638.3±40.7 MPa. Yield strength and strain changed to 16 ±0.8 MPa and 4.4±0.4 %, respectively. Tensile strength increased approximately 100 % above that of soy-PE fibers to 84.2±16 MPa as seen in Figure 4-19. This increase is a proof of a better interphase between matrix and soy flour. However, the modulus did not improve much, but the literature studies have shown that a poor interphase does not necessarily increase modulus, unlike strength [33]. Furthermore, the strain at break decreased, as expected, to 280±29 % (relative to pure PE). However, in comparison with 20-20-60 and 30-20-50 fibers, 23-7-70 fibers displayed a good improvement in tensile strength that is shown in Figure 4- 19. The modulus and yield point did not show a significant difference compared with those properties of Soy-PE fibers.

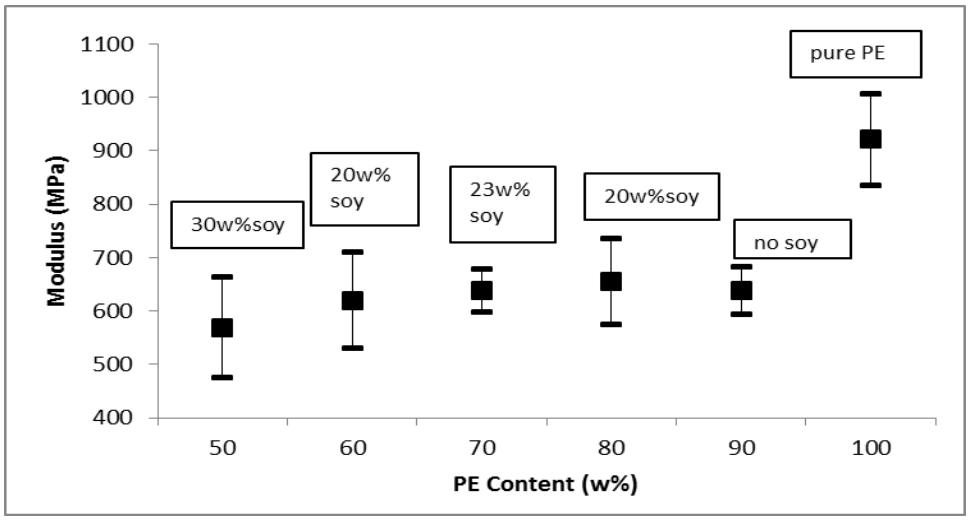


Figure 4- 16 Effect of the PE content on the tensile modulus of soy composite fibers. Soy content is shown beside each data-point, with the remainder being compatibilizer.

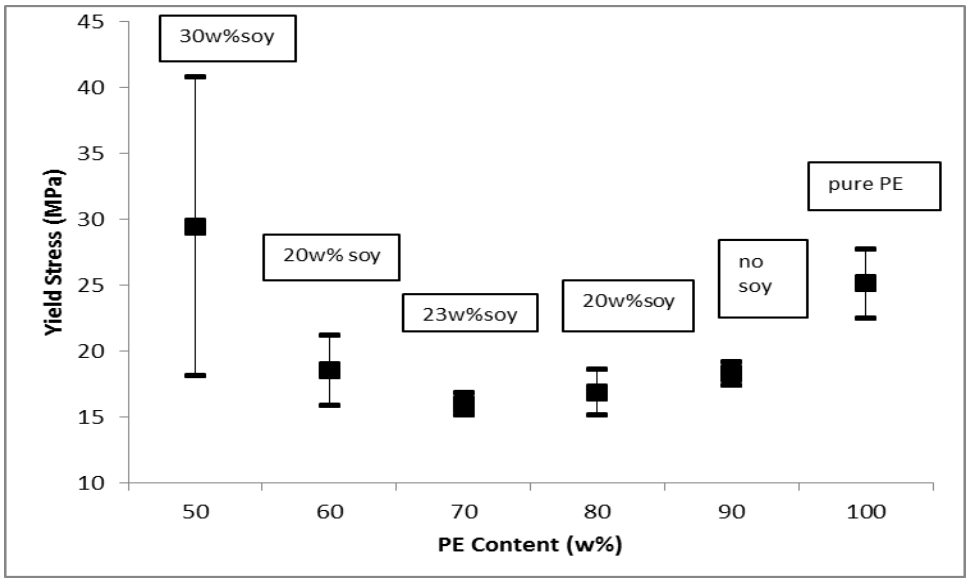


Figure 4- 17 Effect of the PE content on the yield strength of soy composite fibers. Soy content is shown beside each data-point, with the remainder being compatibilizer.

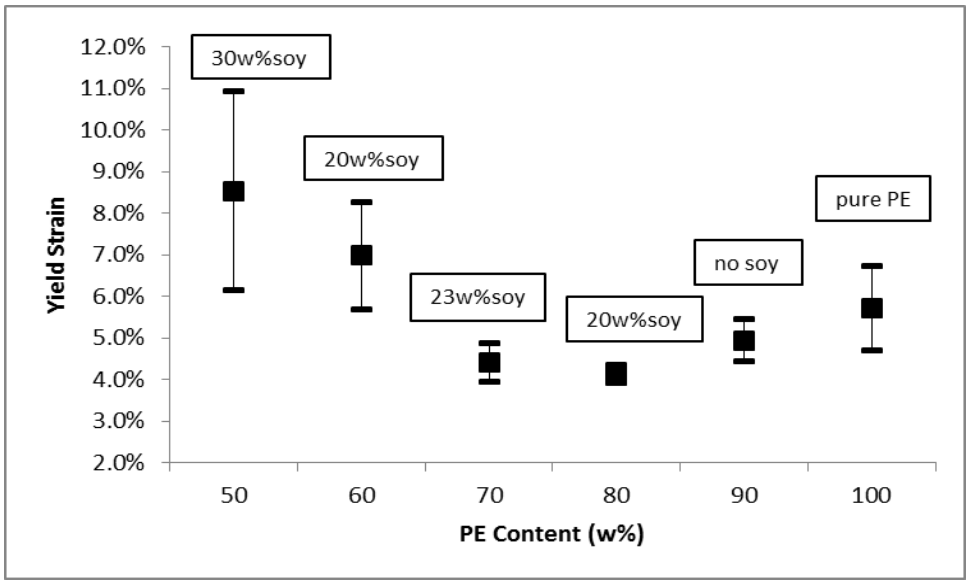


Figure 4- 3 Effect of the PE content on the yield strain of soy composite fibers. Soy content is shown beside each data-point, with the remainder being compatibilizer.

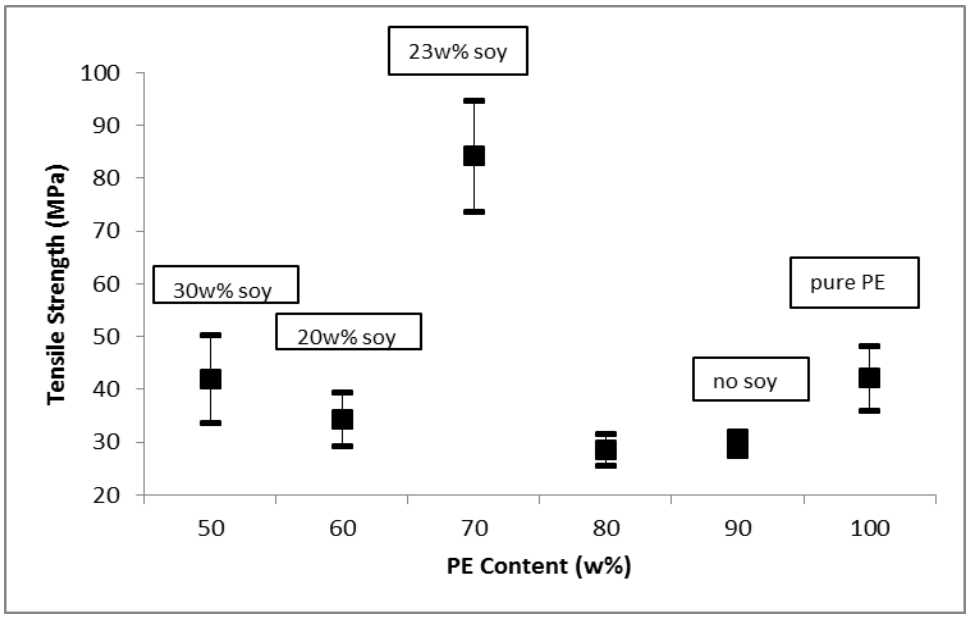


Figure 4- 19 Effect of the PE content on the tensile strength of soy composite fibers. Soy content is shown beside each data-point, with the remainder being compatibilizer.

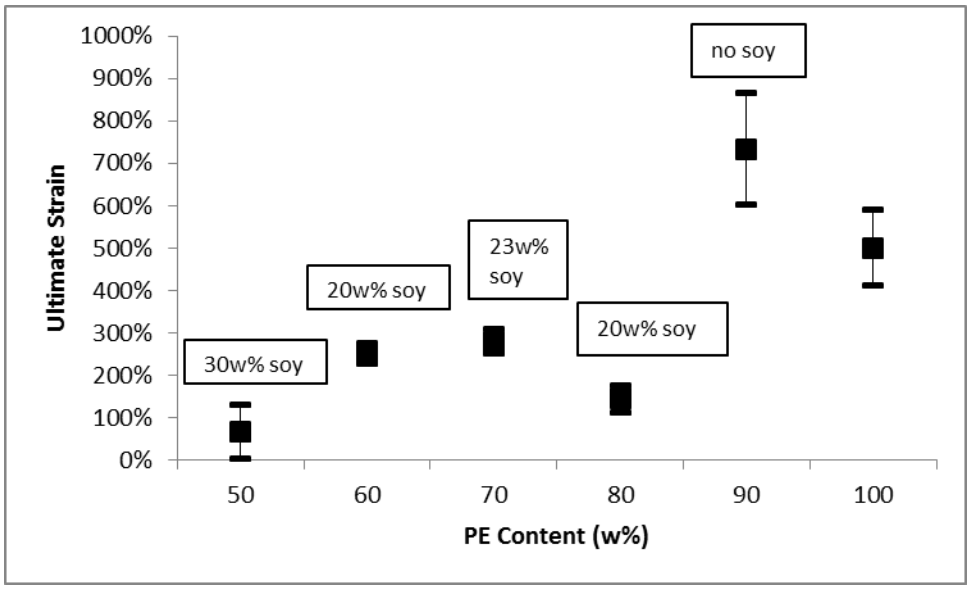


Figure 4- 20 Effect of the PE content on the ultimate strain of soy composite fibers. Soy content is shown beside each data-point, with the remainder being compatibilizer.

As a result, the microstructure improved while soy (filler) content of fibers with compatibilizer decreased. Thus, pure PE has better mechanical properties than the composite fibers. However, 23-7-70 fibers had the best microstructure among soy-modified fibers, and they had the highest tensile strength among such fibers.

To test the size effect on mechanical properties, 23-7-70 fibers were processed with three different draw-down ratios. Tensile modulus was measured at 697±106 MPa, 638±41 MPa and 528±96 MPa for average diameters of 45, 55 and 65 μm, respectively. The yield strains were 5±1 %, 4.4±0.5 %, and 4.4±1 % whereas ultimate strains were 325±38 %, 280±30 %, and 264±70 % for average diameter of 45, 55, and 65 μm,

respectively. Thus, tensile modulus, yield strain and ultimate strain decreased slightly with increasing diameter, but the reduction was not statistically significant. When the average diameter was 45 μm, the yield strength was measured at 19±1.6 MPa and tensile strength at 112±31 MPa. By increasing diameter to 55 μm, yield strength decreased to 16.0±0.8 MPa, and tensile strength to 84.2±10.7 MPa. Then, for a larger fiber diameter of 65 μm, yield strength and tensile strength were measured at 12.1±1.6 MPa and 52±15 MPa, respectively. Thus, yield strength and tensile strength decreased significantly as diameter increased. Therefore, the processing conditions also affect the mechanical properties of fibers.

Long- Term Properties

Aging

After two months of aging at ambient conditions, the tensile modulus for non-aged fibers was measured at 638.2±40.7 MPa, which not change significantly relative to the unaged fibers. However, the modulus changed between two months aged and four months of aging; it slightly decreased to 561±104 MPa at four months of aging. After six months, the fiber tensile modulus was measured at 513.1±48 MPa. Yield stress was measured at 16.1±0.8, 18±3, 15±2 MPa and 13.7±2.4 MPa for non-aged, two-month aged; four-month aged fibers, and six-month-aged fibers, respectively. A significance reduction was observed between non-aged and six-month aged fibers, but not between unaged and others. Moreover, after two months, the only property that changed was the tensile strength, which reduced by approximately half to 35.4±15.2 MPa. The tensile

strength of fibers showed a significantly decreasing trend after six month aging, as shown in Figure 4-21. The non-aged fibers had an average tensile strength of 84.2±16 MPa, which was higher than that of 25±1.4 MPa for six month aged fibers. In addition, the strain at yield and break remained unchanged after six month aging. Strain at yield was 4.4±0.5 %, 4.3±0.6 %, 5.6±0.9 % and 5.1±0.4 % for non-aged, two-month-aged, four-month-aged and six-month-aged fibers, respectively. On the other hand, strain at break was measured at 279.8±29.4 %, 187±68.5%, 308±19.1% and 287.5±29.5% for non-aged, two-month-aged, four-month-aged and six-month-aged fibers, respectively.

As a result, it could be inferred that aging weakened the modulus, yield stress, and tensile strengths; but it did not affect the yield and ultimate strain. The decrease in tensile strength during aging is a consistent result with those reported by Cunningham et al [3] for SPI-based films; however, the strain result is not. It should be noted that SPI-based film samples did not have a PE matrix, and were not really composites.

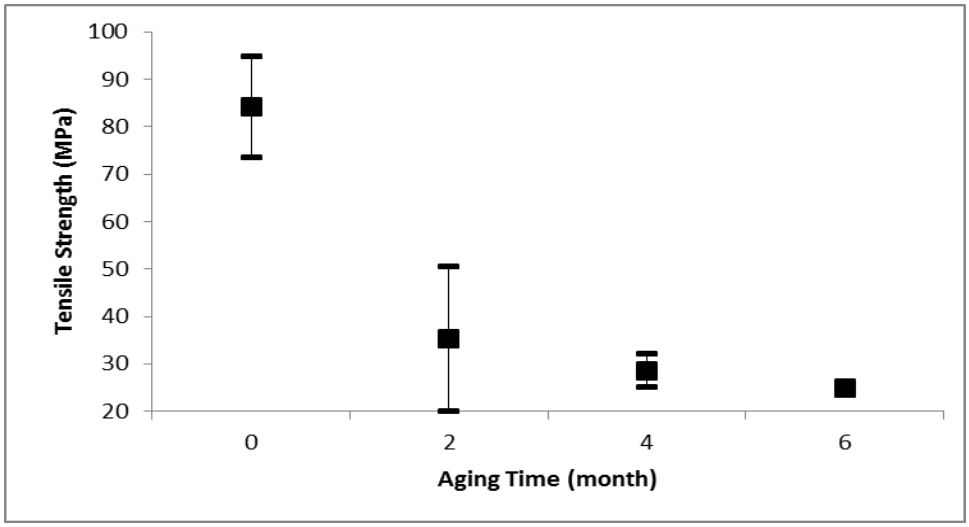


Figure 4- 21 Effect of the aging on the tensile strength of soy composite fibers

Boiling

The tensile modulus for 23-7-70 fibers was 541.1±104 MPa for 10-min-boiled fibers and 580.4±128.3 MPa for 100-min-boiled fibers. Yield stress was at 15±2 for 10-min-boiled fibers, and at 14.2±1.7 MPa. It showed a slight reduction, but the change was not statistically significant (95% confidence level). Yield strain was almost similar for non-boiled, 10-min-boiled and 100-min-boiled fibers at 4.4±0.5 %, 4.8±0.6 %, and 4.4±0.7 %, respectively. Ultimate strain was recorded as 280±29.6 %, 271.6±25.9 %, and 242.2±36.1 % that was similar for non-boiled, 10-min-boiled and 100-min-boiled fibers. The only significant decrease was detected in the tensile strength. After 10 min boiling, it decreased to 27.7 ±4.4 MPa from 84.2±10.7 MPa; and after 100 min boiling, it became to 25±4 MPa as shown in Figure 4-22. A noticeable difference could be seen when fibers were boiled for 10 min, but further boiling did not affect much the tensile strength much more. Mechanical tests showed that there were not any significant difference between modulus, yield strength, strain at yield and strain at break between non-boiled, 10-min boiled and 100-min boiled fibers.

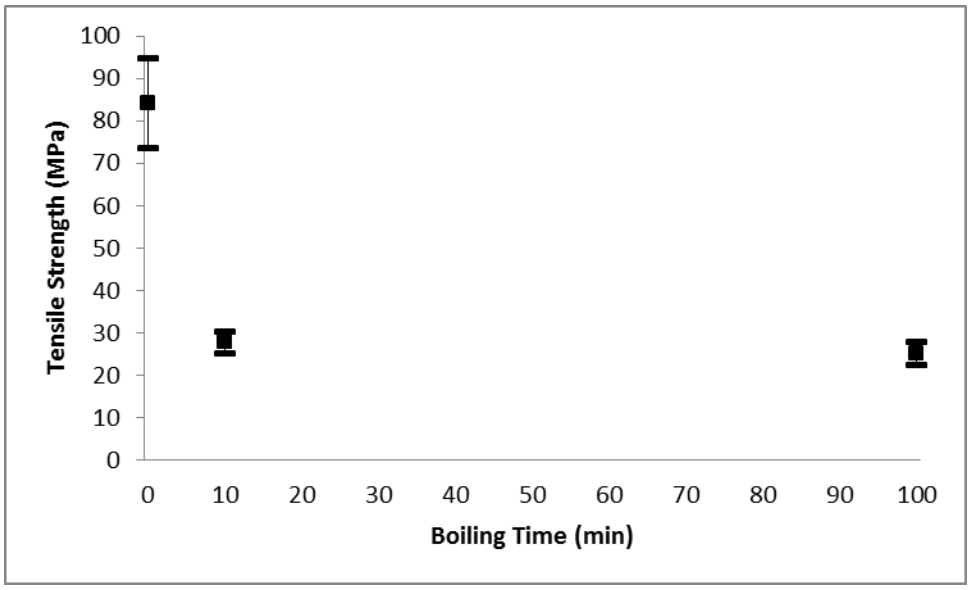


Figure 4- 22 Effect of boiling time on tensile strength of fibers

As a result, the only mechanical property of fibers affected by boiling was tensile strength. It reduced during boiling. The microstructure on the fiber surface was also affected due to the dissolved soy particles from the surface. The decrease in the tensile strength is inferred by the voids formed on the fiber after boiling.

CHAPTER 5

CONCLUSIONS AND FUTURE RECOMMENDATIONS

The primary objectives of this study were to investigate the mechanical and physical properties of fibers made of soy flour, compatibilizer (C) and linear low density polyethylene (PE) as a function of the composition, size and microstructure. Another objective was to investigate the variation in properties of the fibers as a function of aging and environmental effects. This chapter summarizes the conclusions drawn from results presented in Chapter 4. At the end, recommendations for future work are presented.

The results of this study led to the following conclusions:

- Pure PE and C-PE fibers had very smooth surfaces, which became rough after soy particles were added. Without compatibilizer, soy-PE fibers had the roughest surface with large soy agglomerates oriented along the fiber direction. The soy particles on 23-7-70 fibers had a more homogenous distribution and better dispersion (less agglomerates) than other fibers. They had the best microstructural properties of various soy-based fibers investigated in this study.
- Tensile modulus, yield stress, yield strain, tensile strength and ultimate strain of 23-7-70 fibers were 638.3±40.7 MPa, 16±0.8 MPa, 4.4±0.4 %, 84.2±16 MPa, and 280±29 %, respectively. The fibers have moderate mechanical properties that one likely suitable for textile applications (advantage is their lower cost relative to pure PE fibers).

- After ambient aging, tensile strength of the fibers decreased from 84.2±10.6 MPa to 35.4±15.2 MPa, 28.6±3.1, and 24.2±1.1 MPa after two-month, four-month, and six-month aging, respectively; while other mechanical and physical properties were not significantly affected. Aging resulted in a decrease in tensile strength after two months, but there was no significant change in it after next four months. These results suggest that after initial decrease, these fibers are suitable to store at ambient conditions for several months. When aged by accelerated boiling, tensile strength of non-aged fibers decreased to 25±4 MPa after 100 min boiling while the other mechanical properties did not change significantly. Boiling also affected the microstructure of fibers due to the dissolution of protruded soy particle present on fiber surfaces, which led to a smoother fiber surface, but with the presence of small voids. Over-washed fibers had a lower strength at break, which indicates washing will affect the properties of composite soy fibers. However, the tensile strength of washed fibers has still moderate tensile strength as compared with the other textile fibers.

The following recommendations are suggested for future studies:

- Tensile analysis of fibers should be performed at different temperatures and humidity levels to investigate their effect on the mechanical properties.
- Finite element modeling could be used to model their tensile moduli.
- To confirm that inexpensive bio-composite soy fibers have potential applications in textile industry, other properties of fibers should be explored such as impact strength, UV and heat resistance.

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